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Nemati Giv, Ali ; Rastegar, Sina ; Özcan, Mutlu

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Influence of nanoclays on water uptake and flexural strength of glass–polyester composites

Ali Nemati Giv¹ , Sina Rastegar¹ and Mutlu Özcan²

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Abstract

Fiber-reinforced polyester composites have received significant attention in a variety of applications due to their considerable potential due to such characteristics as high strength, stiffness, and modulus. However, one of the most important concerns about polymeric composites is their sensitivity to moisture attack. This work has been conducted to investigate the effects of nanoclay addition on reinforcing glass/polyester composites against water absorption and the resultant deterioration of flexural strength. Therefore, chopped strand mat and woven fiberglass polyester specimens were fabricated by using the hand lay-up technique with varying weight percentages of Cloisite 20A nanoclays (0, 1.5, and 3 wt%) and immersion in water for a time duration of 21 days. The specimens were weighed for the water absorption test. The results showed a remarkable drop in water absorption of the composite samples with the increase of nanoclay content. Moreover, although all the pure and nanocomposite specimens underwent degradation in flexural strength due to the water absorption, the strength was found to significantly increase with increasing the percentage of nanoclay at all immersion periods. The experimental results were confirmed by scanning electron microscopy (SEM). SEM images indicated that the presence of nanoclay protected the fiber/matrix interfaces.

Keywords

Fiber-reinforced polymer composite, nanoclay, water absorption, flexural strength

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Introduction

In recent years, nanocomposites have attracted interest for many applications because of their superior properties compared to traditional composites. Weight, cost, electrical performance, and various mechanical properties (such as stiffness, strength, and fracture toughness) can be improved depending on the nano-sized reinforcements added to the composite materials.^{1–4}

Several studies have been carried out to investigate the effect of the addition of nano fillers made of particles, fibers, or sheets on the mechanical performance improvement of the host polymer.^{5–12} For example, the mechanical properties of composite material made of a hybrid phenol-formaldehyde polymer reinforced with glass and oil palm fibers were studied by Sreekala et al.¹³ Tensile and flexural strength and the tensile modulus showed a great enhancement by addition of glass fibers into the polymer, whereas the hardness of glass-reinforced polymer reduced.

One of the main concerns in using fiber-reinforced polymer composites is the interaction nature of environmental factors, such as water and moisture, with these kind of materials. It is well known that when polymer nano-composites (PNCs) are immersed in water or exposed to moisture, moisture gradually penetrates to the polymer and may adversely affect the properties of the composite.^{14–16} Katnam et al.¹⁷ studied the effect of

¹School of Mechanical Engineering, Iran University of Science and Technology, Tehran, Iran

²University of Zurich, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland

Corresponding author:

A Nemati Giv, School of Mechanical Engineering, Iran University of Science and Technology, University St., Hengam St., Resalat Square, Narmak, Tehran, 16846, Iran.
Email: a.nemati-giv@tu-braunschweig.de



moist environment on the static and fatigue behaviors of rubber-toughened epoxy adhesive. Results indicated that both the static and fatigue resistances of adhesively bonded joints are considerably affected by the moisture absorption.

Nano filler reinforcement has been recognized as one of techniques for the prevention of moisture ingress in polymer composites. Adding nanoparticles to the polymeric materials can reduce the susceptibility of the composite to water or moisture attack. Velmurugan and Manikandan¹⁸ showed that the addition of glass fiber with palmyra fiber into the polymeric matrix reduces the moisture absorption. Many researchers have investigated the influence of harsh environments on the mechanical properties of PNCs. Ishai¹⁹ reported the effect of hot and cold water on the longitudinal tensile strength of glass fiber-reinforced epoxy composites. The study showed 29% and 3% loss in the tensile strength after weathering in hot and cold environments, respectively. Todo et al.²⁰ investigated the effects of moisture absorption on the interlaminar fracture toughness of carbon/epoxy composites. Results indicated that the dynamic interlaminar fracture toughness reduces by 22.7% in the environmental conditions with temperature and relative humidity of 80°C and 90%, respectively. The moisture absorption of an epoxy reinforced with carbon nanotubes (CNTs) and carbon nanofibers (CNFs) with weight percentages (wt%) of 0.25 and 0.5 was analyzed by Gude et al.²¹ They observed that adding the CNTs and CNFs decreases the moisture uptake by 9.29% and 10.4%, respectively, in a 95% relative humidity environment at 55°C. They also reported that the resin diffusion coefficient is not affected by CNFs, whereas adding CNTs reduces the diffusion coefficient by 33%. Khoramishad and Alizadeh²² studied the effects of multi-walled carbon nanotubes (MWCNTs) and silicon carbides (SiCs) on the degradations of mechanical properties of the epoxy polymer immersed in the distilled water. The results showed that the epoxy strength degradation dropped by 32% and 16% by adding MWCNTs and SiCs into the epoxy, respectively, at the saturation point. Meanwhile, the adhesive stiffness degradations of the epoxy reinforced with MWCNTs and SiCs were 27% and 19%, respectively.

According to the literature, researchers have conducted extensive research on polymer nanocomposites. However, there are few studies on polyester resin-based nanocomposites. Polyester resins are widely being used in marine applications because of their ease of handling, low price, and providing an acceptable balance of mechanical, electrical, and chemical properties.²³ A concern with using polyester-based composites in marine applications is degradation of the polymer matrix and fiber/matrix interphase by a hydrolysis reaction of unsaturated groups within the polyester resin, which may reduce the mechanical properties.^{24,25}

In this research, the effects of adding BYK Cloisite 20A nanoclay were investigated on the water absorption and flexural strength of glass/polyester composites. After fabricating pure composites and nano-reinforced composites samples, the laminates were immersed in distilled water and the water absorption was measured for each period of time. The mechanical behavior of the samples was also measured using flexural testing.

Experimental details

Composite materials

The polymer used in this study was an orthophthalic polyester resin (Synolite 1035-T-1) supplied by Dulux Australia with a density of 1.10 g/cm³. The glass/polyester laminates were made using two types of glass fibers (emulsion chopped strand mat and woven glass fiber provided by Colan Products PTY Ltd) with areal masses of 225 and 120 g/m², respectively. Nanoclay Cloisite 20A purchased from BYK USA Co. was used as reinforcement to investigate the amount of water absorption and consequently the degradation of flexural strength of glass/polyester resin reinforced with Cloisite 20A clay nanoparticles. The density of the clay plate nanoparticles was 1.80 g/cm³.

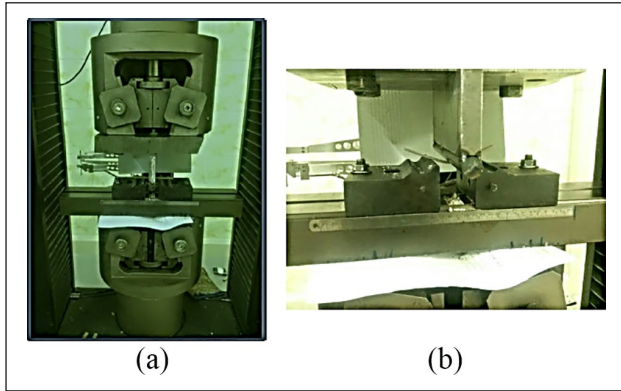
Sample manufacturing

E-glass composites were prepared using the hand lay-up technique and, in order to obtain smooth surfaces on the top and bottom of the composite sheets, two glass plates were utilized as a mold for the preparation of composites. Also, wax and film separators with the brand names of Sika pasty wax 818 and RENLEASE QZ-5101, respectively, were used, which cause the separation process from the glass plates to be performed easily. The designed lay-up for preparation of the desired composite consists of nine layers with a stacking sequence of C/C/W/C/W/C/W/C/C (C: chopped strand mat E-glass fiber and W: woven E-glass fiber). After adding an accelerator and catalyst to the polyester resin according to their standard ratio, the mixture was added to E-glass lay-ups step by step by suitable brushes and uniformly spread over the entire sheet by a laminating roller kit. To ensure a proper curing process, the fiberglass composite samples were allowed to cure at room temperature for 2 days.

In order to investigate the effects of nanoparticles on the amount of water ingress and flexural strength of the E-glass/polyester composite, nanocomposite samples were prepared. Nanocomposite samples were fabricated by a mixing ratio of 1.5 and 3 wt% of Cloisite 20A nanoclay into the resin. To uniformly distribute nano reinforcements, sonication and mechanical stirring were considered as the dispersion methods. At the first stage, nanoclays were added to the polyester resin according to the determined value and

Table 1. Sample specifications according to ASTM D 570 and ASTM D 790 for water absorption and bending tests.

Parameters for sample	Sample for water absorption test	Sample for bending test
Length	76.2	127
Width	25.4	12.7
Thickness	3.2	3.2

**Figure 1.** Three-point bending tests: (a) during the test; (b) after fracture.

then the mixture was stirred using a Remi RQ-5 Plus mechanical stirrer with a speed rate of 1500 rpm for at least 20 minutes to assure the homogeneity of the mixture. After the homogenization process by a mechanical stirrer, the mixture was placed into a Bandelin SONOPULS HD 3200 homogenizer with a power of 100 watts and 2 second on/off cycle for 20 minutes.

Testing of samples

The samples were cut out from the composite sheets by a water jet cutting machine and prepared for measuring the amount of water absorption and flexural strength as per the standards ASTM D 570 and ASTM D 790, respectively. The dimensions of the samples are presented in Table 1. In order to measure the aforementioned properties, five samples were considered for each time period of immersion. For each determined time interval (7, 14, and 21 days), the specimens were weighed by a scale with an accuracy of 0.1 mg to monitor mass changes.

Three-point bending tests of specimens were done using a GALDABINI universal testing machine. The bending specimens were tested with a displacement rate of 1.5 mm/min until the failure point occurred for each specimen (Figure 1). Flexural strength (σ_f) of the specimens was calculated using following equation

$$\sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

in which P is the peak load from the load–displacement curve, L is the gauge length, b is the width, and d is the thickness of the specimen.

Results and discussion

Water absorption

In order to investigate the effects of nanoclay reinforcements on the amount of water absorption of glass/polyester composites, the mass of the composite samples was weighed in both the dry and immersed states after 3, 7, and 21 days. The weight percentage of water absorption ($W\%$) was determined using the following equation

$$W\% = \frac{W_t - W_0}{W_0} * 100 \quad (2)$$

where W_t is the weight of the aged sample after immersion time t and W_0 is the initial weight of the sample before immersion. The variation of water absorption percentages of the glass/polyester samples without nanoparticles and with weight percentages of 1.5% and 3% of nanoclays are represented in Table 2.

According to Table 2, the average water absorption percentages of pure composite samples are 1.28%, 1.58%, and 1.79% for the immersed days of 3, 7, and 21, respectively. Also, these water absorption percentages for the samples reinforced with 1.5 wt% of nanoclays are 1.01%, 1.44%, and 1.62% and regarding 3 wt% of nanoclays are 0.88%, 1.35%, and 1.51%. Figure 2 illustrates the variation of water absorption changes of pure composite samples with Cloisite 20A nanoclay-reinforced composites over different immersion days.

As can be seen in Figure 2, water absorption increases with increased immersion time for all cases. However, water absorption rates decreased over time. The polymer composite materials at the end stages of prolonged immersion time have different behavior so that the weight percentage of the polyester composite reduces because of the chemical reaction of the molecules of polyester and water.^{26,27} From Figure 2, it is also observed that water absorption decreases by adding nanoclay. The maximum average water absorption occurred for the pure composites, whereas the 3% nanoclay composite sample has the lowest average value under similar conditions.

Bending test

Another parameter affected by adding nanoparticles is the flexural strength of the composite. When composites are kept in water or moist environments, the diffused moisture gradually changes their mechanical properties. Similar to the water absorption tests, nanocomposite samples with weight percentages of 1.5% and 3% of nanoclays were

Table 2. The water absorption percentages of the (a) pure composite, (b) 1.5% nanocomposite, and (c) 3% nanocomposite samples.**(a).**

Immersion time (days)	Water absorption percentage of samples	Average water absorption percentage	Standard deviation	Coefficient of variation (CV%)
3	1.20	1.28	0.07	5.47
	1.29			
	1.23			
	1.15			
	1.33			
7	1.56	1.58	0.03	1.90
	1.58			
	1.59			
	1.52			
	1.60			
21	1.81	1.79	0.03	1.68
	1.83			
	1.78			
	1.85			
	1.78			

(b).

Immersion time (days)	Water absorption percentage of samples	Average water absorption percentage	Standard deviation	Coefficient of variation
3	1.03	1.01	0.04	3.96
	0.95			
	1.04			
	1.01			
	1.02			
7	1.51	1.44	0.08	5.56
	1.35			
	1.38			
	1.43			
	1.53			
21	1.58	1.62	0.03	1.85
	1.61			
	1.66			
	1.62			
	1.62			

(c).

Immersion time (days)	Water absorption percentage of samples	Average water absorption percentage	Standard deviation	Coefficient of variation
3	0.86	0.88	0.04	4.55
	0.90			
	0.91			
	0.82			
	0.91			
7	1.3	1.35	0.09	6.67
	1.28			
	1.34			
	1.5			
	1.31			
21	1.51	1.51	0.04	2.65
	1.49			
	1.58			
	1.47			
	1.50			

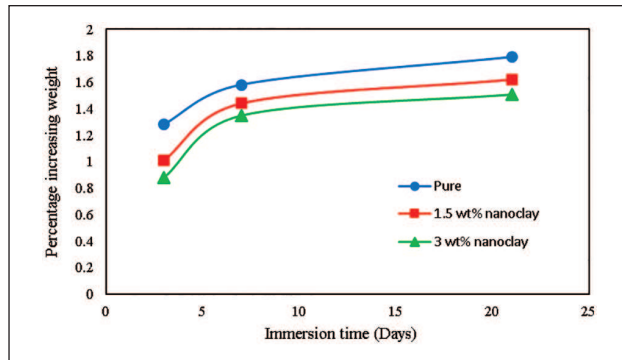


Figure 2. Water absorption versus immersion time for the pure and Cloisite 20A nanoclay-reinforced composite samples.

considered for comparison with pure composite specimens. The removal times of 7 and 21 days were selected for the bending tests. In order to investigate the flexural properties of the glass/polyester samples, five specimens for each state were tested. After testing, the flexural strength of each sample was determined using a load–displacement graph of the tested samples. The results of the bending test are tabulated in Table 3.

It is clearly seen in the Table 3 that all specimens experienced strength reduction during water immersion as a result of the detrimental effects of moisture on the mechanical behaviors of the composite. Figure 3 illustrates the average flexural strength of the composites unreinforced and reinforced with nanoclay particles with various weight

Table 3. The flexural strength of the (a) pure composite, (b) 1.5% nanocomposite, and (c) 3% nanocomposite samples. (a).

Immersion time (days)	Flexural strength of samples (MPa)	Average strength (MPa)	Standard deviation	Coefficient of variation
0	185.00	183.96	8.04	4.37
	172.20			
	194.68			
	182.53			
	185.39			
7	158.90	164.00	4.44	2.71
	164.14			
	168.75			
	160.21			
	168.00			
21	148.91	150.83	3.56	2.36
	153.45			
	155.36			
	146.46			
	150.07			

(b).

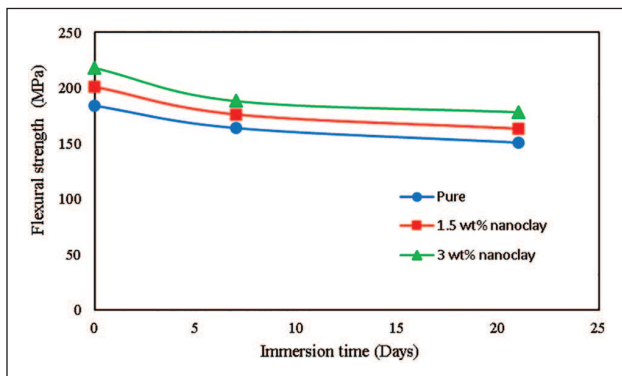
Immersion time (days)	Flexural strength of samples (MPa)	Average strength (MPa)	Standard deviation	Coefficient of variation
0	202.25	200.91	3.92	1.95
	203.37			
	196.10			
	197.55			
	205.28			
7	175.57	176.04	3.13	1.78
	179.86			
	172.68			
	178.61			
	173.48			
21	161.89	163.12	2.15	1.32
	162.84			
	165.64			
	160.26			
	164.65			

(Continued)

Table 3. (Continued)

(c).

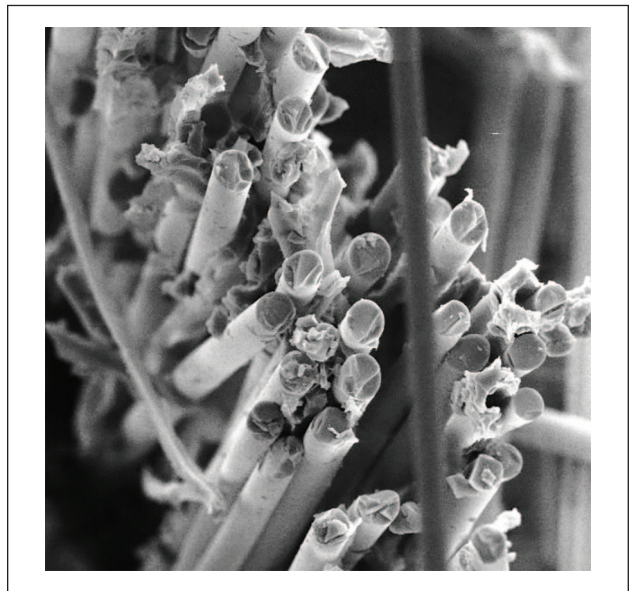
Immersion time (days)	Flexural strength of samples (MPa)	Average strength (MPa)	Standard deviation	Coefficient of variation
0	214.58	218.42	4.90	2.43
	216.56			
	222.36			
	224.82			
	213.78			
7	188.40	188.41	2.52	1.34
	185.03			
	191.69			
	189.73			
	187.20			
21	177.47	178.30	3.96	2.22
	179.35			
	183.53			
	172.51			
	178.66			

**Figure 3.** Flexural strength immersion time for the pure and nanoclay-reinforced composites.

percentages over different time intervals. Each datum in Figure 3 is based on the average results of five specimens.

As is obvious in Figure 3, adding nanoclay into the glass/polyester composite results in improvement in the flexural strength for all aging conditions so that higher weight percentages of nanoclay particles showed higher flexural strength.

Broken sections of the glass/polyester specimens obtained from the bending test after the water immersion were observed using a scanning electron microscope. Scanning electron microscopy (SEM) images of composite samples without nanoclay immersed in undistilled water for the time period of 21 days are shown in Figure 4. As can be seen in Figure 4, the surfaces of some glass fibers are smooth without polymer particles, which may provide evidence of debonding at the fiber/matrix interphase. This was because of the moisture absorption of the interphase. In fact, voids and microcracks present in the

**Figure 4.** Scanning electron microscopy images of glass-fiber-reinforced polymer samples without nanoclay after 21 days of water immersion.

polymer can trap water molecules, which cause damage to the interphase and strength degradation.

Figure 5 illustrates the SEM images of the fiber-reinforced polyester composites containing 3% of Cloisite 20A after 3 weeks of immersion time. SEM analysis indicates suitable bonding in glass/polyester interfacial area in which many portions of polyester resin have still stuck to the fibers even after 21 days of immersion (see in Figure 5). This is due to the fact that the nanoclay is hydrophilic and can absorb moisture and, therefore, the fiber/matrix interface

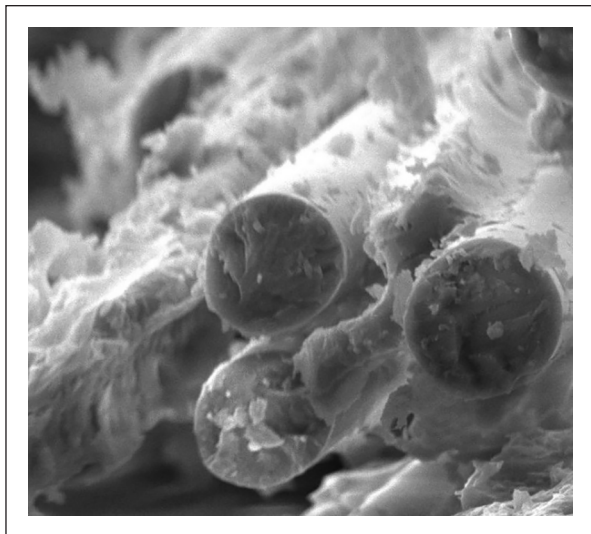


Figure 5. Scanning electron microscopy images of glass-fiber-reinforced polymer samples containing 3% of nanoclay after 21 days of water immersion.

would interact with fewer water molecules, resulting in little damage to the nanocomposite sample.

Conclusion

When the glass/polyester composites are immersed in water, the water absorption of the hydrophilic groups in the glass fiber and the polyester affects the properties of the composites. Adding nanoparticles to polymer composites is a method of reinforcing composites against the water attack, which can reduce the water absorption and resultant degradations of mechanical properties. This paper presents the fabrication of nanoclay-reinforced glass/polyester composites with weight percentages of 1.5 and 3 of Cloisite 20A nanoclays. The water absorption experiments revealed that the water weight percentages of polymer composites dropped by adding nanoparticles at different stages of water absorption. In addition, the dry and wet specimens were tested under bending loads and the flexural strength of the composites were determined. According to the results, a remarkable reduction in flexural strength of composite samples was shown by increasing the immersion time. The results of the bending tests showed that 3 wt% nanoclay and the pure composite have the highest and lowest flexural strength, respectively.

Declaration of conflicting interests

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ORCID iD

A Nemati Giv  <https://orcid.org/0000-0002-1618-804X>

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